



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1623c

Sulfur in Residual Fuel Oil

This Standard Reference Material (SRM) is intended for use in the calibration of instruments and the evaluation of methods used in the determination of total sulfur in fuel oils or materials of similar matrix. The certified sulfur content is based on analyses using isotope dilution thermal ionization mass spectrometry (ID-TIMS) [1]. Homogeneity testing was performed using X-ray fluorescence spectrometry and no significant bottle to bottle variability was observed. The certified value for the sulfur content, reported as a mass fraction expressed in percent, is given below. A unit consists of 100 mL of commercial “No. 4 (light)” residual fuel oil as defined by reference 2, ASTM D 396-95 Standard Specification for Fuel Oils [2], in an amber bottle.

Certified Sulfur Content: 0.3806 % \pm 0.0024 %

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where u_c is the combined standard uncertainty calculated according to the ISO Guide [3] and k is a coverage factor. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with material inhomogeneity and ID-TIMS measurement uncertainty. In the absence of Type B uncertainties (which are negligible here in comparison with Type A), the expanded uncertainty (U) given is for a 95 % prediction interval. The coverage factor, $k = 2.31$, is Student's t -value for a 95 % prediction interval with eight degrees of freedom.

Expiration of Certification: The certification of this SRM is valid until **13 June 2012**, within the uncertainty specified, provided the SRM is handled in accordance with instructions given in this certificate (see “Instructions for Use”). However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

Stability: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any substantive changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by W.R. Kelly, R.D. Vocke, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division.

Analytical measurements were performed by W.R. Kelly, R.D. Vocke, A.F. Marlow, P.A. Pella, and J.R. Sieber of the NIST Analytical Chemistry Division.

Statistical calculations for this SRM were carried out by S.D. Leigh of the NIST Statistical Engineering Division.

The support aspects involved in issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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Gaithersburg, MD 20899
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See Certificate Revision History on Last Page

INSTRUCTIONS FOR USE

Handling: The SRM bottle should only be opened for the minimum time required to dispense the material. To relate analytical determinations to the certified value on this Certificate of Analysis, a minimum sample mass of 140 mg should be used. After use, the bottle should be tightly capped and stored under normal laboratory conditions away from direct sunlight

SUPPLEMENTAL INFORMATION

The physical properties of SRM 1623c are listed in the table below. These properties were determined using ASTM methods by a commercial firm under contract to NIST. The results are **NOT** certified and are provided as additional information on the matrix.

| Physical Property Test | ASTM Standard | Result |
|-----------------------------|------------------------|--|
| Density @ 15 °C | D 1298-85 (1990) | 899.6 kg/m ³ |
| @ 60 °F | D 1250-80 (1990) | 25.7 API |
| Flash Point, PMCC | D 93-94 | 52 °C |
| Pour Point | D 97-93 | 21 °C |
| Heat of Combustion, Gross | D 240-92 ^{e1} | 43.98 MJ • kg ⁻¹ (18 907 Btu • lb ⁻¹) |
| Kinematic Viscosity @ 40 °C | D 445-94 ^{e1} | 5.015 • 10 ⁻⁶ m ² /s (5.015 cSt) |
| @ 50 °C | D 445-94 ^{e1} | 4.002 • 10 ⁻⁶ m ² /s (4.002 cSt) |
| @ 100 °C | D 445-94 ^{e1} | 1.801 • 10 ⁻⁶ m ² /s (1.801 cSt) |
| Carbon | D 5291-92 | 87.8 % |
| Hydrogen | D 5291-92 | 11.7 % |

ASTM Standards Used

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|--------------------------------|---|
| D 1298-85 (1990) ^{e1} | Standard Practice for Density, Relative Density (Specific Gravity) or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method |
| D 1250-80 (1990) ^{e1} | Standard Guide for Petroleum Measurement Tables |
| D 93-94 | Standard Test Methods for Flash Point by Pensky-Martens Closed Tester |
| D 97-93 | Standard Test Methods for Pour Point of Petroleum Products |
| D 240-92 ^{e1} | Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter |
| D 445-94 ^{e1} | Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity) |
| D 5291-92 | Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants |

REFERENCES

- [1] Kelly, W.R.; Paulsen, P.J.; Murphy, K.E.; Vocke, R.D., Jr.; Chen, L.T.; *Determination of Sulfur in Fossil Fuels by Isotope Dilution-Thermal Ionization Mass Spectrometry*, Anal. Chem., Vol. 66, pp. 2505-2513 (1994).
- [2] D 396-95; *Standard Specification for Fuel Oils*; Annu. Book of ASTM Stand., Vol. 05.01 (1995).
- [3] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; ISO: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.

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| Certificate Revision History: 05 May 2004 (This revision reflects an extension of the certification expiration date); 13 June 2000 (This revision reflects a change in the certification expiration date); 30 July 1996 (Original certificate date). |
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Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.